

IOP Conference Series: Materials Science and Engineering

PAPER • **OPEN ACCESS**

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To cite this article: N Dudova *et al* 2021 *IOP Conf. Ser.: Mater. Sci. Eng.* **1014** 012010

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Evolution of strengthening factors during long-term aging at 650 °C in advanced 10% Cr heat-resistant steel

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Abstract. The effect of long-term aging for 1000...~40,000 h at 650 °C on the tensile strength at ambient temperature was studied in a low-nitrogen and high-boron 10%Cr martensitic steel. In order to establish the reason for increasing the yield stress and ultimate tensile strength after 10,000 h of aging, the evolution of strengthening factors was analyzed. A decrease in the substructure and solid solution strengthening during long-term aging is compensated by an increase in the dispersion strengthening due to the precipitation of V-rich MX carbonitrides.

1. Introduction

New generation fossil power plants with higher thermal efficiency and reduced emission of carbon dioxide require the heat-resistant materials that operate at higher temperatures. The most heated components of the power units will be made of austenitic steels and nickel-based superalloys. Nevertheless, the 9-12% Cr martensitic steels will be the base materials due to their excellent combination of high creep strength, good fatigue and oxidation resistance, and low cost [1]. One of the advanced approaches to improve the 100,000 h creep strength up to a minimum value of 100 MPa at 650 °C is a steel alloying modification by increasing the B content and decreasing the N content [2,3].

The creep resistance of these steels is associated with the stability of non-equilibrium structure, which is called the tempered martensite lath structure (TMLS). The complex TMLS consists of prior austenite grains, packets, blocks and laths with a high dislocation density in the lath interiors [2,3]. The main dispersion strengthening phases are nanoscale boundary $M_{23}C_6$ -type carbides and MX carbonitrides homogeneously distributed in the laths. The role of precipitates of secondary phases ($M_{23}C_6$, MX, Laves phase) in the high stability of TMLS under creep condition is the subject of research interest. It was recently revealed that long-term aging led to a slight increase in the yield strength and ultimate tensile strength [4]. The aim of this work is to examine the effect of long-term aging at 650 °C on the evolution of strengthening factors in advanced 10% Cr steel with low N and high B contents at ambient temperature.

2. Experimental

A 10% Cr steel with the following chemical composition (in wt.%) 0.1C, 0.06Si, 0.1Mn, 10.0Cr, 0.17Ni, 0.7Mo, 0.05Nb, 0.2V, 0.003N, 0.008B, 2.0W, 3.0Co, 0.002Ti, 0.006Cu, 0.01Al and Fe-balance was examined. The vacuum induction-melted steel was subsequently hot-forged by Lasmet, Chelyabinsk. The steel was subjected to standard heat treatment: normalization at 1060 °C/30 min and tempering at 770 °C/3 h. Small specimens for tensile tests were cut from the grip portions of creep tested specimens



at 650 °C, subjected only to long-term thermal aging for 1000; 10,000; 28,286 and 39,437 hours [4]. Tensile tests were carried out using flat specimens with a gauge length of 4 mm and cross-sectional dimensions of 1 mm x 1 mm using an Instron 5882 testing machine at ambient temperature and a strain rate of $\sim 10^{-3} \text{ s}^{-1}$. The structural characterization was performed using a Jeol JEM-2100 transmission electron microscope (TEM) with an INCA energy dispersive X-ray spectrometer. The transverse lath widths were measured on TEM micrographs by the linear intercept method. The dislocation densities were estimated by counting individual dislocations in the (sub)grain/lath interiors per unit area on at least six arbitrarily selected typical TEM images for each data point.

3. Results and discussion

3.1. Tensile strength and microstructure after long-term aging

Aging of the 10% Cr steel at 650 °C for 1000...~30,000 h leads to a slight increase in the yield strength (YS) and ultimate tensile strength (UTS) (Figure 1a). Maximum rise of YS and UTS (+3...6%) to 520 and 695 MPa, respectively, occurs for 10,000 h aged steel. A ~40,000 h aging results in the UTS of 650 MPa that is 4...6% lower as compared with the tempered condition, whereas the YS returns to the initial level of 500 MPa. Therefore, the 10%Cr steel obviously demonstrates higher tensile strength characteristics after aging at 650 °C for 10,000 h.

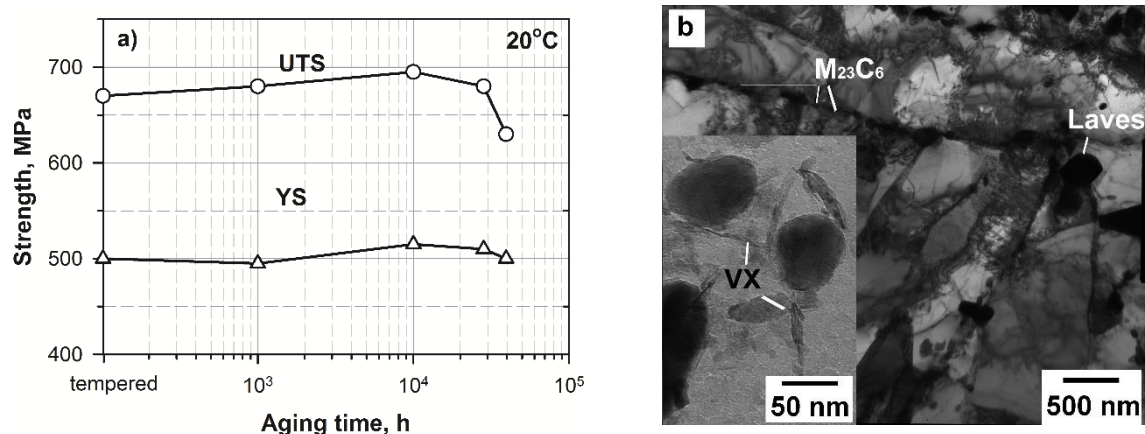


Figure 1. Effect of long-term aging at 650 °C on the YS and UTS at 20 °C (a). Microstructure of the 10% Cr steel after long-term aging for 10,000 h at 650 °C (b).

The TMLS of the 10% Cr steel is stable under long-term aging condition at 650 °C (Figure 1b) and does not transform into the subgrain structure up to ~40,000 h. Lath width increases less than 2 times (Table 1). Dislocation density in the lath interiors continuously decreases by 4 times.

Secondary phases in the tempered steel are presented by nanoscale M₂₃C₆ carbides (70 nm) located on the boundaries and fine Nb-rich MX carbonitrides (30 nm) homogeneously distributed in the laths. Both M₂₃C₆ and MX precipitates are resistant to coarsening. An insignificant coarsening of M₂₃C₆ carbides from 70 nm to 96 nm after ~40,000 h occurs. Nb-rich MX are highly stable.

Aging leads to Laves phase (Fe₂(W,Mo)) precipitation at the boundaries (Figure 1b). Consequently, depletion of W and Mo from the ferritic matrix occurs (Table 1). The onset of the Laves phase coarsening appears after 1000 h and accelerates after 10,000 h.

Between 1000 and 10,000 h of aging, fine V-rich MX carbonitrides precipitate in the lath interiors (Figure 1a). The mean size of these particles estimated by TEM observation of carbon replicas is 26.5 nm. The volume fraction of V-rich MX phase is 0.00862% as calculated by Thermo-Calc. However, structure observations reveals approximately 0.1% of V-rich MX particles.

Table 1. Change in the structural parameters of the 10% Cr steel during long-term aging at 650 °C.

Structural parameters		Duration of aging (h)				
		0	1000	10,000	28,286	39,437
Dislocation density, $\times 10^{14}$ (m^{-2})		1.70	1.23	1.06	0.61	0.43
Lath width (μm)		0.38	0.409	0.507	0.566	0.614
Concentration of element in the matrix (at. fraction):						
	Cr	0.109	0.105	0.104	0.103	0.1004
	W	0.0062	0.0034	0.003	0.0026	0.0024
	Mo	0.004	0.0034	0.0029	0.0024	0.0018
	Co	0.0304	0.0304	0.0304	0.0304	0.0304
Mean size of particles (nm) ^a :						
	M_{23}C_6	70	72	81.9	83.6	96
	Laves phase	-	145.6	197.6	298.8	319
	NbX	30	35	31.3	31.6	35
	VX	-	-	26.5	40	58

^a The volume fraction of M_{23}C_6 is 2.048 / 2.107%, Laves – 0/1.6%, NbX – 0.05/0.06%, VX – 0/0.1% (before / after aging, respectively.)

3.2. Strengthening factors

To reveal the reason for the increase in the steel's strength after long-term aging for 10,000 h, the strengthening factors were analyzed. Estimation was carried out in accordance with a model describing the YS of high-chromium steels [5,6]. The YS is presented as:

$$\sigma_Y = M \sqrt{(\tau_A^2 + \tau_B^2)} \quad (1)$$

where M is the Taylor factor (2.9), τ_A is the strengthening from dislocations, τ_B is the strengthening from obstacles.

Dislocation strengthening is determined as: $\tau_A = \alpha_1 \mu b \sqrt{\rho}$, where α_1 is a constant (0.2); μ is the temperature depending shear modulus (at 20 °C it is 83 GPa); b is the Burgers vector ($2.48 \cdot 10^{-10}$ m); ρ is the dislocation density.

Strengthening from the obstacles can be defined as a sum of the strengthening from Peierls-Nabarro barriers (τ_{PN}), low angle boundaries (τ_{BD}), solute atoms (τ_{SS}) and dispersion particles (τ_{prec}) estimated according to Ref. [5,6]: $\tau_B = \tau_{PN} + \tau_{BD} + \tau_{SS} + \tau_{prec}$. Dispersion strengthening is determined by the Orowan-Ashbi model as [5,7]: $\tau_{prec} = 0.045(\mu b / \lambda) \ln(r/b)$, where λ is the mean distance between particles, r is the mean radius of particles.

The estimated strengthening factors are shown in Figure 2 (a-c). The evolution of strengthening during long-term aging can be described as follows:

- the dislocation strengthening continuously decreases with aging time, however, in the range of 1000...10,000 h the intensity of decreasing is the smallest;
- the solid solution strengthening by substitutional atoms of Cr, W, Mo, and Co is slightly reduced due to precipitation of W- and Mo-rich particles of Laves phase;
- the substructural strengthening by low angle boundaries of martensitic laths is continuously decreased starting from 1000 h of aging due to the lath widening;
- the dispersion strengthening during aging for 1000...10,000 h is increased on 16% as compared with tempered condition. Although the strengthening from M_{23}C_6 and Laves phases is reduced, precipitation of V-rich MX particles compensates for this decrease.

The calculated values of YS (Figure 2d) remain stable between 1000 and 10,000 h of aging and are close to the experimental YS. This fact is resulted from the increasing precipitation strengthening.

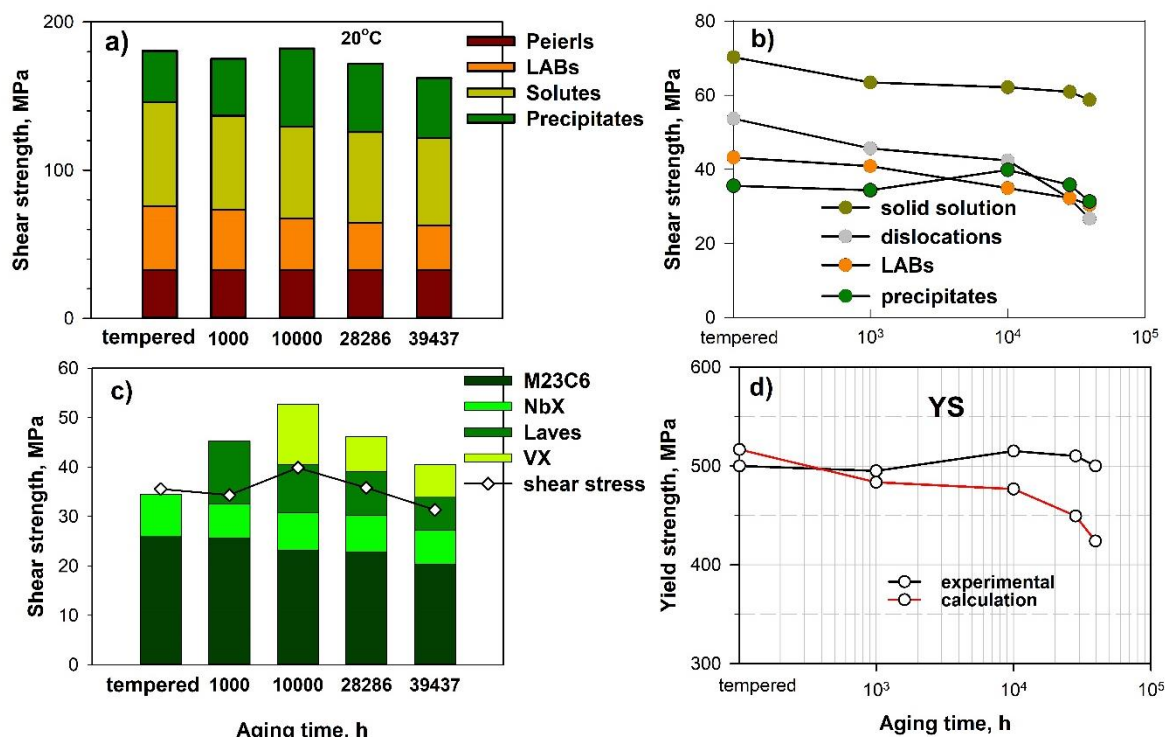


Figure 2. Change in the strengthening factors (a-c) during long-term aging at 650 °C of the 10% Cr steel. Change in the calculated YS at 20 °C after aging in comparison with experimental data (d).

4. Conclusion

The stabilization of the calculated yield strength of the low-nitrogen and high-boron 10% Cr steel is revealed after 1000...10,000 h aging at 650 °C that is attributed to an increase in dispersion strengthening. Precipitation of fine V-rich MX carbonitrides during aging for 1000...10,000 h can contribute to the strengthening of this steel.

Acknowledgments

The authors are grateful to the staff of the Joint Research Center, “Technology and Materials”, Belgorod State University, for providing the equipment for instrumental analysis.

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